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DOI:
10.3933/ApplRheol-28-42903

Applied Rheology | DOI: 10.3933/APPLRHEOL-28-42903 | WWW.APPLIEDRHEOLOGY.ORG

Viscoelastic Properties of Gallium-indium Alloy

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Received: 8.2.2018, Final version: 8.6.2018

Abstract:
The viscoelastic properties of a gallium-indium alloy in the pre-yield region make it easier to understand their characteristics, particularly the varying degrees of stiffness and damping properties. These viscoelastic properties were measured with a strain-controlled rheometer, where both strain amplitude sweep mode and the angular frequency sweep mode were conducted. Three groups of experiments were carried out in the strain amplitude sweep mode. In the angular frequency sweep mode, the storage modulus G' and the loss modulus G'' were investigated at the linear region, the critical region, and the non-linear region. Experimental results indicate that the gallium-indium alloy exhibited similar viscoelastic properties. The linear viscoelastic region takes place when the strain amplitude is less than the critical strain amplitude of 1 %. At the critical regime, the gallium-indium alloy has the strongest relative elasticity. These results are helpful to understand the intrinsic properties of gallium-indium alloys and find their application in flexible circuits, soft robotics, self-healing, and mechanical shock absorption.

Key words:
Viscoelasticity, gallium-indium alloy, storage modulus, loss modulus

1 INTRODUCTION

Liquid metals, especially gallium-indium alloys, are attracting researchers' considerable interests because such materials exhibit liquid characteristics at room temperature and good thermal conductivity, which makes them useful for soft robotics, the electricity industry, micro channels and surface, and within the medical field [1–4]. Compared to mercury, gallium-indium alloy is virtually non-toxic, and has a similar surface tension but with a higher thermal conductivity, which enables it to have a wide range of applications as well as safe use in the laboratory and in industry [5, 6]. For instance, many attempts have recently been made to apply gallium-indium alloy to various research and industrial fields by utilizing its quick response and easy control characteristics.

Viscoelastic properties have undergone extensive research in fields such as magnetorheological (MR) fluids and MR elastomers [7–9], but to broaden the application of gallium-indium alloy its material properties must be quantified, particularly its viscoelasticity. Eustathopoulos and Drevet [10] investigated surface tension by experimenting with the flow of liquid metal in micro-channels, but the resulting measurements have limited usefulness for understanding the viscoelastic properties of the oxide skin of liquid metal in air. Gallium-indium alloy can be used as sacrificial 'inks' for fabricating 2D and 3D microfluidic channels [11]. Larsen et al. [12] conducted an in-depth study of the viscoelasticity of liquid metal oxide films and their results demonstrated that oxides of Ga on liquid metal substrates in atmospheric conditions exhibit a complex variety of mechanical behavior. Chossat et al. [13] invented a novel soft strain sensor where two liquid conductors: an ionic solution and a eutectic gallium-indium alloy are placed into a microchannel, while Bartlett et al. [14] generated high thermal conductivity in soft elastomers with elongated liquid metal inclusions that consist of the gallium-indium alloy and a soft elastomer. Their sensor demonstrated relatively high accuracy and reliability when measuring large strains. However, if more research into the viscoelasticity of gallium-indium alloy is carried out the mechanical properties of their senor can be better applied. While some research has been carried out using this low-melting-point alloy vary its stiffness [15, 16], further research would enable the characteristics of variable stiffness to be quantified.

The viscoelastic properties of liquid metals, particularly gallium-indium alloys are important because they help us understand the mechanical properties, even though the viscoelastic properties are needed for flexible robots based on gallium-indium alloy, as well as for constructing flexible circuits and simulating liquid metal. Whereas most existing work is aimed at the application of gallium-indium alloy there are only a few reports that explain its properties, especially viscoelasticity.

In this work we investigated the viscoelastic properties of gallium-indium alloys with properties ranging from 95 % gallium, 5 % indium, to 50 % gallium and 50 % indium. The viscoelastic properties of this alloy include the storage modules G', the loss modules G'', and the loss factor $\tan \delta = G''/G'$. A strain controlled rheometer modified by Anton Paar was used to investigate the sample. The effects of the strain amplitude γ_o and angular frequency ω on the viscoelastic parameters were examined using the strain amplitude mode and frequency sweep mode measurements. We also defined and identified parameters such as the critical strain and critical frequency. The remainder of this paper is as follows: Section 2 provides the linear and non-linear viscoelastic theories which form the basis of these tests. Section 3 plans the experimental steps, the gallium-indium alloy samples, and the equipment needed for the experiments. Section 4 expounds the experiments carried out on various samples in sweep mode and frequency sweep mode. The results of experiments are presented and discussed in detail in Section 5 and Section 6 concludes the paper.

2 LINEAR VISCOELASTIC THEORY AND NON-LINEAR VISCOELASTIC THEORY

2.1 LINEAR VISCOELASTICITY

Various methods are used to measure viscoelastic properties of materials, of which the small-amplitude oscillatory shear test is the most widely used. Many studies on the pre-yield rheology of materials are based on linear viscoelastic theory, so Dynamic Mechanical Analysis (DMA) [14, 15] was used to measure the linear viscoelastic properties of gallium-indium alloy. However, as a viscoelastic material, gallium-indium alloy can produce stresses in the same phase and out of phase components.

$$G^* = G' + iG'' \tag{1}$$

where G' is the storage (in-phase) modulus and G'' is the loss (out-of-phase) modulus. The phase angle δ indicates whether the material is more like a solid or a liquid, therefore a useful quantity called the material loss factor may be defined from the complex modulus

$$\tan\delta = \frac{G''}{G'} \tag{2}$$

The loss factor is a measure of the ratio of the energy consumed by the material per radian during a steady sinusoidal excitation to the stored energy.

2.2 NON-LINEAR VISCOELASTICITY

Gallium-indium alloy has linear viscoelastic properties at a small enough strain range, so in the linear viscoelastic region, the complex shear modulus G^* depends only on the frequency ω. However, in the nonlinear viscoelastic region, the shear modulus depends on the frequency ω and the strain amplitude so the linear viscoelastic equation can no longer describe the dynamic behavior of that region [17]. The stress response of gallium-indium alloy in large-amplitude oscillatory shear is not sinusoidal, so if the fluid inertia is negligible, the shear stress in the alloy can be represented as a Fourier series of odd harmonics

$$\tau(t) = \sum_{m=1,odd}^{M} \tau_m \sin(m\omega t + \delta_m) \tag{3}$$

where τ_m is the amplitude and δ_m the phase content with both depending on the strain amplitude γ_o and the frequency ω. Each Fourier component is separated into an in-phase part and an out-of-phase part and a one-dimensional constitutive equation of the following form can be produced as follows:

$$\tau(t) = \sum_{m=1,odd}^{M} \left[G'_m \gamma_o^m \sin(m\omega t) + G''_m \gamma_o^m \cos(m\omega t) \right] \tag{4}$$

G_m' and G_m'' are the m-th storage modulus and loss modulus, respectively. By combining Equations 3 and 4 we can obtain the first harmonic storage modulus and loss modulus, which can be expressed as:

$$G'_1 = \frac{\tau_1}{\gamma_o} \cos(\delta_1) \tag{5}$$

$$G''_1 = \frac{\tau_1}{\gamma_o} \sin(\delta_1) \tag{6}$$

3 EXPERIMENTAL SETUP

3.1 PREPARING THE SAMPLE

The alloy sample has proportions ranging from 95 % gallium and 5 % indium to 50 % gallium and 50 % indium 50 % as listed in Table 1. While the melting point varies with the proportion of gallium-indium alloy, the melting point of alloy has a minimum value of 14.2°C where the proportion is 85 % gallium and 15 % indium. When the alloy has a fusion ratio which occupies a large proportion of the metal, its melting point is closer to the melting point of the metal. When preparing this alloy we found that when the sample has 65 % gallium and 35 % indium, or close to it, the liquid metal no longer appears to be liquid, it has a non-Newtonian fluid state similar to mud, even if the room temperature is lower than its melting point. A non-Newtonian fluid state is an intermediate state between solid and liquid.

3.2 EXPERIMENTAL EQUIPMENT

An advanced rheometer with a parallel-plate geometry that can work in stress and strain controlled modes was used to measure the viscoelastic properties of the gallium-indium alloy. The characteristics and dimensions of the plate to plate configuration for gallium-indium alloy are radius of the rotor plate R = 9 mm, width of gap h = 0.15 mm, and volume of gallium-indium alloy in the measuring gap V = 0.25 ml. The rheometer's measuring system has four main parts: A suction pump to prepare the rheometer, a parallel-plate to test the samples, and a temperature regulation tank with water and a control computer. The gallium-indium alloy sample was placed in a constant gap of 0.15 mm so that the oscillating shear strain signal can be compared to the shear stress signal. In strain-controlled mode a sinusoidal strain with a given amplitude and frequency is applied to the sample and then the torque or stress is measured and the storage modulus G', loss modulus G'',

Gallium [%]	Indium [%]
95	5
90	10
85	15
80	20
75	25
70	30
65	35
60	30
55	45
50	50

Table 1: The samples of gallium and indium alloy.

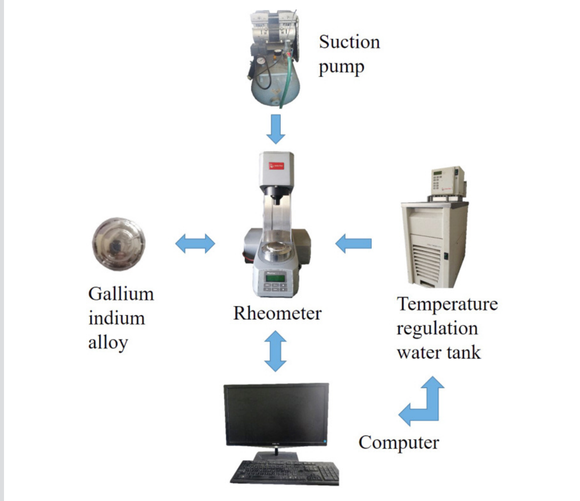

Figure 1: Schematic of the rheometer.

and loss factor tan $\delta = G''/G'$ are calculated from the relationship between the applied signals and response signals [19]. This advanced rheometer can alter its operating temperatures from 5 to 50°C, including the melting temperature of all the samples, to meet the requirements of our experiments. The schematic of the rheometer was shown in Figure 1.

3.3 DYNAMIC MEASUREMENTS

Measurements are taken by comparing the oscillating shear strain signal with the response shear stress signal and then applying the sine strain of a given amplitude, the frequency, and the resulting stress with the controlled strain rheometer. The dynamic characteristics are then calculated from the relationship between the applied signal and the response signal. The disk edge shear strain amplitude γ_0 was calculated by

$$\gamma_o = \frac{\varphi_o R}{h} \tag{7}$$

where φ_0 is the angular amplitude of oscillation, R is the 9 mm radius of the rotor, and h is a constant gap of 0.15 mm. The rheometer's measuring system and software is then used to obtain the storage shear modulus G', the loss modulus G'', and loss factor tan $\delta = G''/G'$ as the function of the oscillatory frequency ω and strain amplitude γ_0.

4 RESULTS

The viscoelastic properties of gallium-indium alloy with various proportions of gallium and indium, and the strain amplitude and driving frequency were investigated. In this experiment, the control variable method was used to obtain the effect of a single parameter of the gallium-indium alloy. The strain amplitude γ_0 swept

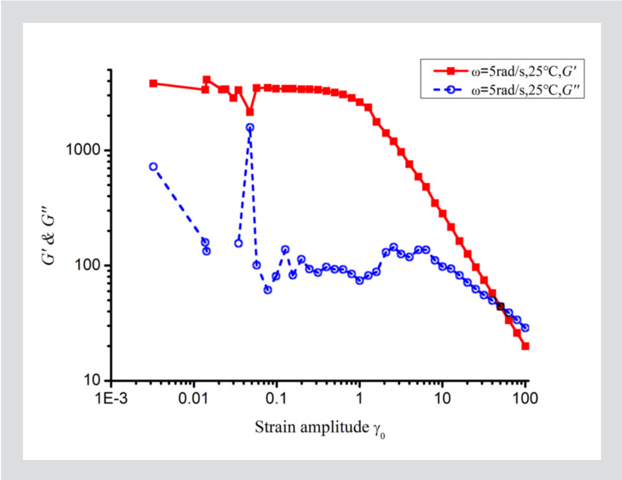

Figure 2: An example of the storage modulus and loss modulus LVE region in strain amplitude swept mode.

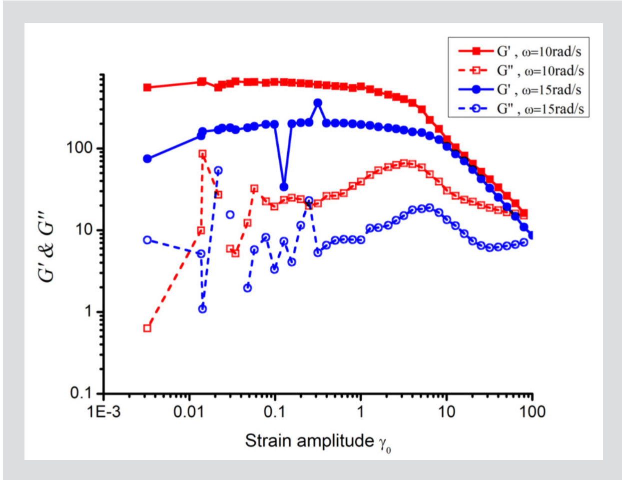

Figure 3: The storage modulus and loss modulus of 85 % gallium and 15 % indium at a temperature of 25°C and with frequencies of 5, 10, and 15 Hz.

from 0.01 to 100 % and driving frequency swept from 1 to 100 Hz. The temperature was set to vary from 5 to 50 °C according to the melting point of each proportion of gallium-indium alloy.

4.1 AMPLITUDE OF THE SWEEP MODE

To determine the amplitude of the strain range over which linear viscoelasticity (LVE) holds, we carried out strain sweep experiments at a frequency of 5 rad/s over a range of strain amplitudes of 0.01 ~ 100 %. The gallium-indium alloy exhibited linear viscoelastic properties in the pre-yield region [20, 21], so if the strain amplitude γ_o is smaller than the yield strain γ_{lin}, the alloy sample exhibits linear viscoelasticity, however, a critical strain γ_{lin} may be approximately determined using the following condition:

$$\frac{G'(\gamma_o = \gamma_{lin}, \omega)}{G'(\gamma_o \to 0, \omega)} = 90\% \tag{8}$$

This allows for a 10 % tolerance of the initial value. Figure 2 is an example of linear viscoelasticity. The sample was 85 % gallium and 15 % indium alloy under an Amplitude sweep mode at 20°C. Within this LVE region the storage modulus G' of the alloy reaches 3500 Pa, whereas in the non-linear viscoelastic region the storage modulus G' decreased sharply as the strain amplitude increased. Figure 2 shows that G' at a strain amplitude of 100 % is almost 3 orders less than the value in the LVE region, which means the energy storage capability of the liquid metal decreases sharply as the strain amplitude increases. A plot of the loss modulus G'' versus the strain amplitude γ_o is shown in Figure 2: Here the loss modulus tended to stabilize at strain 0.1. It then increases slightly before passing through a maximum and then decreasing as the strain amplitude increased. Figure 2 also can reflect the loss factor tan δ as a function of the strain amplitude γ_o. In the region where the strain amplitude is 0.1 to 1, the loss factor is so small that the gallium-indium alloy exhibits mainly elastic properties with $G' >> G''$, but as the strain amplitude increases, the loss factor gradually increases and the viscous effect of the alloy eventually dominates when the loss factor exceeds 1.0 and $G' > G''$.

We used the amplitude sweep mode in the experiment to achieve the effect of the strain amplitude γ_o on the dynamic properties. The applied strain amplitude was increased from 0.001 to 100 and the driving frequencies were 5, 10, and 15 Hz. The temperature was set to 25°C to ensure that all the samples were liquid during the experiment. Figure 3 shows the storage modulus G' and the loss modulus G'' against the strain amplitude γ_o of the sample with 85 % gallium and 15 % indium at 25°C in 5, 10, and 15 Hz. All these results, as shown in Figures 2 and 3 indicate that the storage moduli begin with a linear trend that decreases as γ_o increases, but when the trend is stable the loss modulus increases as γ_o first increases and then decreases as γ_o increases. However, the loss factor increases as the strain amplitude increases. Note that at the same amplitude, the storage modulus and loss modulus will decrease as the frequency increases, while the loss factor remains basically unchanged. The reason why the storage modulus and loss modulus depend on the amplitude of shear strain can be explained by the internal structure of gallium-indium alloy. Since gallium-indium alloy has a strong surface tension due to the very small shear strain amplitude, it is difficult to destroy the force between the gallium and indium alloy molecules: This suggests that the alloy has dominant elastic properties at very small displacement. When the frequency is 5 Hz, the experimental data obtained is more obvious and the other comparative experiments are complete in a 5 Hz environment.

Figure 4 shows the storage modulus G' and the loss modulus G'' for different ratios of gallium-indium alloy against the strain amplitude γ_o and frequency of 5 Hz. The results show that all the gallium-indium alloy samples exhibited a similar pattern where all the curves had

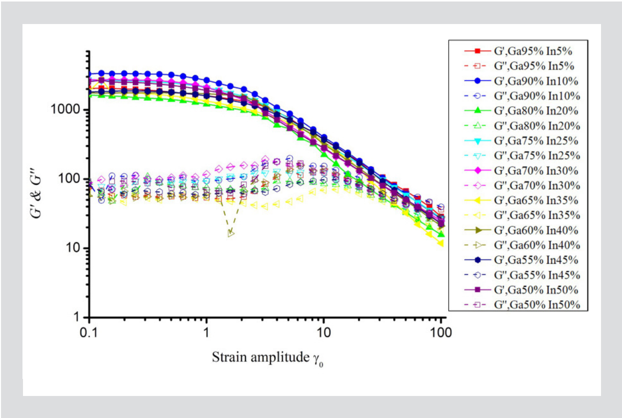

Figure 4: The storage modulus and loss modulus of all the gallium-indium alloy samples at 5 Hz.

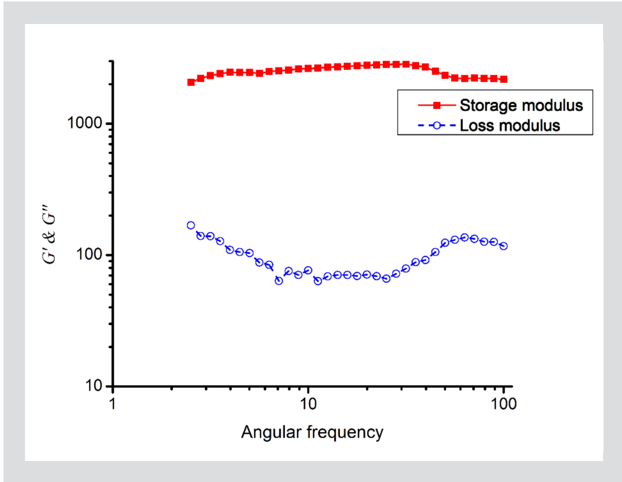

Figure 5: The storage modulus, loss modulus and loss factor in the condition of linear region.

a line trend with a strain amplitude of less than 1, and then the curve of the storage modulus decreased rapidly as the amplitude increased. The loss modulus is independent of γ_o when $\gamma_o \leq 1$. For $1 < \gamma_o < 10$, the curve of loss modulus had a slight increase and then had a sharp decrease when the strain amplitude is above 10, i.e. $\gamma_o \geq 10$. It also can reflect the trend of loss factor. Specifically, for the very small strain amplitudes of $\gamma_o < 1$, it is independent of the strain amplitude, which is obvious because both the storage modulus and loss modulus are constants. In the strain range of $1 < \gamma_o < 10$, the storage modulus started to decrease, while there was a slight increase in loss modulus and in the area $\gamma_o \geq 10$, the storage modulus and loss modulus both showed decreasing trends. Therefore, loss factor had a significant increase at the range of $1 < \gamma_o < 10$ and then increased slowly at the range of $\gamma_o \geq 10$. The results showed that the loss modulus and loss factor of all samples had similar variations: This suggests that regardless of the gallium-indium alloy ratio, in the same state the experimental temperature is higher or lower than the melting point. That shows similar viscoelastic properties in frequencies as low as 5 Hz.

4.2 FREQUENCY SWEEP MODE

The experiments carried out under the frequency dependence of viscoelastic properties of gallium-indium alloy in the linear and nonlinear regions were also analyzed. The effect that the strain amplitude γ_o had on the dynamic properties was established using the frequency sweep method. The critical strain amplitude of all the gallium-indium alloy samples was almost 1 % (shown in Figure 4), which means the storage modulus, loss modulus, and loss factor is approximately a constant in the linear region. Figure 5 shows the effect of frequency ω on the dynamic properties at strain amplitude $\gamma_o = 0.5$ % and the sweep frequency is from 1 to 100 Hz. In this figure the storage modulus G' shows a slight decrease in the region of high angular frequency, whereas the loss modulus G'' increased slightly at high angular frequency. Similarly, the result also shows that the storage modulus is always higher than the loss modulus in that condition, so the curve of the loss factor also has increasing fluctuations in the high angular frequency region. Obviously the loss factor is always less than 1, so the gallium-indium alloy has a predominantly elastic response within frequencies of 1.0 to 100 Hz. Figure 6 shows the condition of G' and G'' at the non-linear viscoelasticity region ($\gamma_o = 10$ %): Here the storage modulus and loss modulus increases with amplitude when the amplitude is less than 10 % and the two parameters decrease sharply at first and then tend to stabiles at the region of non-linear viscoelasticity.

We used the Fourier transform (FFT) analysis [18, 22] to study the nonlinear viscoelastic properties of viscoelastic materials, and to obtain the first-harmonic shear storage modulus G', the loss modulus G'', and the loss factor tan δ. Figure 6 shows the results of 85 % gallium and 15 % indium alloy's storage modulus G', the loss modulus G'', and the loss factor tan δ at strain amplitude where $\gamma_o = 10$ % in the angular frequency sweep mode. The figure shows that in comparison to the linear region, the curve of the storage modulus in the non-limited area is very disorderly. Compared to the storage modulus of the linear region, the storage modulus in the non-linear region decreased more in the high angular frequency region as the angular frequency increased. However, the loss modulus continued to increase until the angular frequency reached 15 Hz, after which it gradually decreased as the frequency increased. Since the loss factor tan δ is the ratio of G'' to G', the law of variation in the low frequency region is similar to the loss modulus, whereas in the high frequency region the loss factor decreases due to a rapid reduction of the gain modulus.

The frequency response of the storage modulus G' and the loss modulus G'' for 85 % gallium and 15 % indium alloy at the linear region (strain amplitude 0.5 %), the critical region (strain amplitude 1 %), and the nonlinear region (strain amplitude 10 %) are shown in Figure 7. Note that the storage modulus at strain amplitudes of 0.5 and 1 %, as the angular frequency increases, the G' at strain amplitudes of 0.5 and 1 % have similar

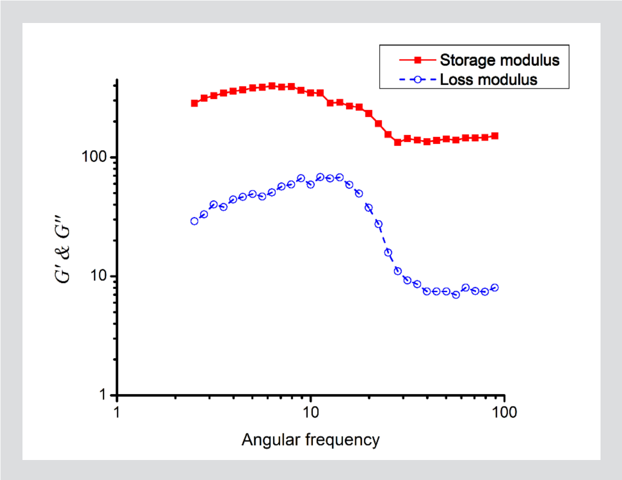

Figure 6: The storage modulus, loss modulus, and loss factor in the non-linear region.

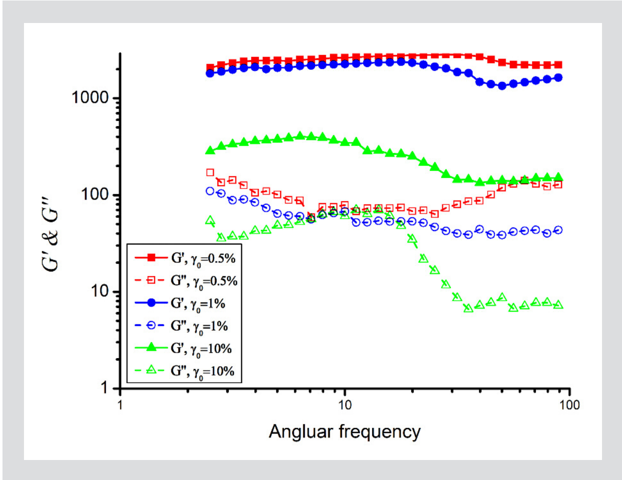

Figure 7: The plots of the storage modulus G' and the loss modulus G'' against the angular frequency ω of the 85 % gallium and 15 % indium sample at 25°C and strain amplitudes of 0.5, 1, and 10 %.

variation rules and approximately the same magnitude, but the storage module in the non-linear region is one-tenth of that in the linear region or critical zone. This suggests that the gallium-indium alloy in the linear region is more rigid and is less prone to deformation. A comparison of the loss modulus in different regions was shown in Figure 7: In the low frequency region, although the curve of each loss modulus is slightly different, the values are similar, whereas in the high angular frequency region, the loss modulus is very sensitive to different regions. Basically, the higher the strain amplitude, the lower the loss modulus. For example, G'' at an amplitude of 0.5 % is more than ten times more than at an amplitude of 10 % in the high frequency region, whereas G'' at an amplitude of 1 % is between an amplitude of 0.5 and 10 %. With the loss factor, the three different group of regions show different trends: Because of G' at an amplitude of 10 % is ten times less than the others while G'' at an amplitude of 10 % is similar to the others in the region when the frequency is less than 20 Hz, the loss factor at an amplitude of 10 % is much higher than the others. Moreover, the loss factor at 1 % is the lowest of the three because G' at an amplitude of 1 % is similar to an amplitude of 10 % and G'' at an amplitude of 1 % is about one-half of that at an amplitude of 0.5 % while G' and G'' at an amplitude of 10 % are almost one-tenth of that at an amplitude of 0.5 %.

5 DISCUSSIONS

All the experimental results are based on the linear and non-linear viscoelastic theory. In the experiments the viscoelastic properties of different proportions of gallium-indium alloy were studied under oscillatory shear using the amplitude sweep mode and frequency sweep mode. Regardless of the proportions of gallium-indium alloy, in the linear region of amplitude ($\gamma_o \leq \gamma_{lin}$), the storage modulus and loss modulus were independent of the strain amplitude, whereas in the nonlinear region with amplitudes from $\gamma_o > \gamma_{lin}$, the storage modulus decreased rapidly as the strain amplitude increased, and the loss modulus first increased and then slowly decreased. Hence, the behavior of gallium-indium alloy ranged from mainly elastic at small strain amplitudes to small viscous at high strain amplitudes. All the samples behaved roughly the same at temperatures above their melting point. This suggests that when gallium-indium alloy is in a liquid state it has almost the same viscoelasticity. This is possibly because when the temperature is higher than its melting point, the molecular structure of gallium-indium alloy is almost the same, so different proportions have almost influence on its viscoelasticity. In a frequency sweep mode and at the linear viscoelasticity region, the storage modulus G' is independent of frequency while the loss modulus G'' had a slight decrease as the frequency increased. In the non-linear viscoelasticity region, the storage modulus G' and loss modulus G'' increased as the frequency increased and then dropped until it stabilized at the high frequency region. The loss factor between the linear region and non-linear region is quite different, i.e. in the linear region the gallium-indium alloy was relatively elastic, and this increased as the frequency increased in the low frequency region, but then the loss factor increased as the frequency increased in the high frequency region until it finally stabilized. However, gallium-indium alloy in the non-linear region was relatively viscous, and became more viscous as the frequency increased in the low frequency region, but in the high frequency region the loss factor decrease and then became stable. Since the critical region of gallium-indium alloy is at an amplitude of 1 %, it is mainly elastic between the low frequency region and the high frequency region because in the low frequency region the storage modulus and loss modulus at an amplitude of 1 % is similar to an amplitude of 0.5 %. In the high frequency region, the storage modulus at an amplitude of 1 % is similar to that at an amplitude of 0.5 % while the loss modulus at an amplitude of 1 % is two times lower than that at an amplitude of 0.5 %. Therefore, gallium-indium alloy at an amplitude of 1 % in any frequency is relatively elastic.

6 CONCLUSIONS

In this paper, the dynamic properties of different proportions of gallium-indium alloys, including the storage modulus G', the loss modulus G'', and the loss factor $\tan\delta = G''/G'$ were studied experimentally. These parameters were measured as a function of oscillating frequency ω and direct strain amplitude γ_0. We found the range of linear viscoelastic strain amplitudes of gallium-indium alloy such that when the strain amplitude was less than γ_{lin} ($\gamma_0 < \gamma_{lin}$), the storage modulus G', loss modulus G'', and loss factor $\tan\delta$ of the gallium-indium alloy is independent of the strain amplitude. Regardless of the proportions of gallium-indium alloy, i.e. the temperature above their melting points the alloy was liquid and its viscoelasticity was roughly the same. By comparing the storage modulus G', the loss modulus G'', and the loss factor $\tan\delta$ in the linear region as well as the critical region and non-linear region with frequency, we found that gallium-indium alloy had the strongest relative elasticity in the critical region. However, the viscoelasticity of gallium-indium alloy in a non-Newtonian fluid state, and whose temperature is close to melting point, and gallium-indium alloy in a solid state whose temperature is lower than the melting point, cannot be measured with a rheometer, so it needs further study.

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